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CERTIFICATION

This is to certify that Corporate Translations, Inc. has performed a true translation for **FMC Corporation** of a patent entitled **"Manufacturing Method of a Capsule Containing Stable Fluid" (Applicant: Snow Brand Milk Products Co., LTD; Application number: JP1973-16183) (CTi Job#FC57927)**. This document was prepared by translators who are bilingual in Japanese and English.

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Date: May 19, 2010

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EXAMINED PATENT GAZETTE

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(54) MANUFACTURING METHOD OF A CAPSULE CONTAINING STABLE FLUID

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DETAILED EXPLANATION OF THE INVENTION

The present invention pertains to the manufacturing of a capsule containing a stable fluid.

To manufacture a capsule with a core that is fluid, conventional methods that have been proposed involve forming a gel film by immersing the gel in fluid state or a liquid comprising a core combined with an alginic acid salt or a low methoxyl pectin in an acid solution or a polyvalent metal salt solution to prepare said capsule. The capsules produced by these methods, however, have had the drawback that the core gels even more or hardens over time and loses its fluidity when stored. After immersing the gel or liquid in an acid solution or a polyvalent metal salt solution to form a gel film by the action of change in pH inhibiting the charge of the free carboxyl of the alginic acid salt or low methoxyl pectin or the action of polyvalent metal ions cross linking the alginic acid salt or low methoxyl pectin thereby continuing to harden and gel over time in said gel or liquid of the core within the film.

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Specifically, because it is of low molecular weight, unreacted polyvalent metal ions on the surface of the gel film formed pass through the gel film and diffuse in the core due to osmotic pressure causing a further cross linking reaction with the alginic acid salt or low methoxyl pectin in the gel or liquid of the core, thus bringing about further hardening or gelling of the gel or liquid of the core.

Moreover, adding sodium phosphate or sodium citrate beforehand to a core sol comprising an alginic acid salt and a low methoxyl pectin has been proposed in order to prevent such gelling or hardening of the core. Contacting a polyvalent metal salt after comprising a core sol of an alginic acid salt and a low methoxyl pectin, however, cannot avoid bringing about a gelling reaction (even if this gelling reaction is minimized to some extent by sodium phosphate or sodium citrate reacting with the polyvalent metal ions), making it impossible to maintain the core as a stable fluid.

The object of the present invention is to eliminate these previous drawbacks by providing a capsule containing a stable fluid in the core that does not lose fluidity over time.

The present inventors found that when they applied a substance that does not react with metal ions as the fluid, such as a gel or a liquid comprising the core of a capsule, and made contact with said sol or liquid combined with a polyvalent metal salt with an alginic acid salt solution or a low methoxyl pectin solution, cross linking occurred between the polyvalent metal ions and the alginic acid salt or low methoxyl pectin in the fluid intended to comprise the core and produced a gelled substance with a three-dimensional network structure. This in turn formed a film on the surface of the fluid, thereby making it so that the fluid in the core enclosed in this film did not gel or harden over time.

This invention which was developed based on this

finding, is characterized by producing a capsule containing a stable fluid by combining a polyvalent metal salt with a fluid substance comprising a gel or a liquid to comprise the core of a capsule, and contacting this blend with a solution of an alginic acid salt, a low methoxyl pectin, or a blend of both to form a gel film.

The configuration and effects of the present invention will be discussed hereinafter.

First, the fluid comprising the core of the target capsule of the present invention is prepared from a gel or a liquid that does not react with metal ions. The materials used to prepare the fluid can be a wide range of gels or liquids that do not react with metal ions, and a variety of additives can be added depending on the use objective of the capsule. For example, if a viscous fluid is desired as the core of the capsule, a natural glutenous substance such as gelatin, agar, or tamarind seed polysaccharide, or a synthetic glutenous substance such as a methylcellulose is used. Additives may be, for example, saccharides, fragrances, or food dye.

Next, the polyvalent metal salt to be added to the fluid comprising a core prepared in this way can be any metal ion that forms a crosslink with an alginic acid salt or a low methoxyl pectin to produce a gelled substance, and is non-hazardous in terms of food hygiene, though a calcium salt such as calcium lactate is especially advantageous. Although not specifically limited, the content of a polyvalent metal salt added to the fluid comprising the core is appropriate at 0.5 to 1.0 wt%. The alginic acid salt is advantageously a sodium salt or a potassium salt, but may also be any salt that is soluble in water.

As noted earlier, the present invention is characterized by contacting a fluid containing a polyvalent metal salt prepared so as to comprise the core of a capsule with a solution of an alginic acid salt or a low methoxy pectin to form a gel film on the surface of the fluid comprising the core. To contact the fluid comprising the core with a solution of an alginic acid salt or a low methoxy pectin, the fluid may be dropped into or immersed in the solution using a depositor. When the fluid comprising the core contacts these solutions, the polyvalent metal salt in the fluid immediately reacts with the alginic acid salt or low methoxy pectin to form a gel film. Although some increase is found in the thickness of the gel film over time, no gelling at all is found of the fluid comprising the core contained in this film. Considering the technical basis for such a phenomenon, it is surmised that after forming a gel film by the reaction discussed earlier, even if unreacted alginic acid salt or low methoxy pectin contacts the gel film, being polymer substances, they do not pass through the gel film to penetrate the core. Unreacted metal ions remaining in the core, however, pass through and diffuse in the gel film due to osmotic pressure, causing gelling on the surface of the gel film which thickens and to some extent hardens the gel film, thus keeping the fluid in the core stable. When a fluid containing a polyvalent metal salt is contacted with an alginic acid salt, the aqueous solution of the alginic acid salt contacting different metal ions from the metal forming this salt forms a gel. Needless to say, care must be taken that the polyvalent metal salt used is of a different metal from the metal forming the alginic acid salt. The thickness and hardness of the gel film forming the capsule can be regulated by adjusting, for example, the concentration of the alginic acid salt or the low methoxyl pectin, the concentration of metal ions, or the time and temperature of the reaction between these. The concentration of metal ions in the fluid comprising the core is adjusted as just noted. A concentration of the solution of alginic acid salt, low methoxyl pectin, or blend of both in a range of 0.5 to 1.0% will form a gel film of suitable hardness.

As discussed earlier, the capsule obtained by the present invention encloses a core substance that is a stable fluid. This capsule itself can impart a distinctive flavor such as in food like jelly, and is very palatable. Such a capsule is spherical, and when mixed with an ice cream product, provides a palatable jelly-filled ice cream product with no foreign body feel because of the thinness of the capsule film.

Various liquid foods can be added to the core of the capsule to provide still more flavorful ice cream products. The present invention can be applied not just

to the production of jelly foods, but also to the production of encapsulated liquid pharmaceuticals and nutritional supplements by hardening the gel film of the capsule.

The present invention will be discussed in more detail with the following working examples.

Working Example 1

Preparation of fluid comprising capsule core:

Gelatin	4 wt%
Granulated sugar	55 wt%
Calcium lactate	2 wt%
2% Color solution	10 wt%
Water	29 wt%

These ingredients were blended well to prepare a sol, which was dropped in spheres of about 5 mm diameter into a 0.5% sodium alginate solution using a depositor. When allowed to rest for about three minutes after dropping, a film of calcium alginate formed on the surface of the sol to encapsulate the sol in spheres. The capsules were separated and washed with water to give the product. The gelatin in the capsules remained a fluid just after encapsulating, and even if gelled several hours later, had weak gel strength. Therefore, fluidity was not entirely lost and the gel strength did not change during storage, thus giving a stable gel capsule.

Working Example 2

Agar	0.5 wt%
Granulated sugar	55 wt%
Calcium lactate	2 wt%
2% Color solution	10 wt%
Water	22.5 wt%

A core substance was prepared from these ingredients as follows. First, just the agar was combined with water and boiled at 100°C to dissolve and prepare a sol. This sol was combined and blended with the other substances, then dropped into a 1% sodium alginate solution at a temperature of about 40°C. Capsules were then obtained following the procedure discussed in Working Example 1. The gel film of the resulting capsule had a harder and thicker gel film than the capsule produced in Working Example 1.

Working Example 3

Preparation of liquid comprising core of capsule:

Gelatin	0.5 wt%
Granulated sugar	55 wt%
Calcium lactate	2 wt%
2% Color solution	10 wt%
Water	22.5 wt%

These ingredients were blended well to prepare a sol, and this blend was dropped into a 0.5% sodium alginate solution. To aid forming spheres of liquid during this dropping, the blend may be combined with a low concentration gelatin solution to increase the viscosity of the solution. Large capsules of 3 to 5 mm diameter were obtained.

Next, the following table shows change in hardness over time in these working examples based on the low dryness condition of 95% RH. A product produced by a conventional method was measured at the same time as a control. To find hardness, the diameter of the capsule was measured beforehand, 50% deformation of the diameter was applied, and the pressure (g) required to produce this deformation was taken as the hardness. Hardness was measured at 20°C and 60% RH.

Time (days)	Working Example 1	Working Example 2	Working Example 3	Conventional Method
0	7.5	11.0	7.5	8.0
1	12.0	15.0	6.0	20.5
2	13.0	17.5	7.5	24.0
3	13.0	17.5	7.5	26.0

Storing in this way for three days revealed the significant difference that where Working Examples 1, 2, and 3 reached a nearly constant hardness in two to three days, the product produced by the conventional method gradually increased in hardness, and continued to rise in hardness thereafter.

(57) **Claim**

1 The Manufacturing Method of capsule containing stable fluid, characterized by contacting a blend of a polyvalent metal salt added to a fluid comprising the core of a capsule with a solution of an alginic acid salt, a low methoxyl pectin, or a blend of both to form a gel film.

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